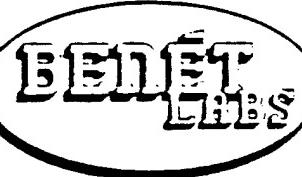# BENÉT INTERNAL TECHNICAL REPORT

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UTILIZATION OF ION CHROMATOGRAPHY AND STATISTICS TO DETERMINE IMPORTANT ACIDS IN CHROMIUM PLATING AND ELECTROPOLISHING SOLUTIONS

SAMUEL SOPOK

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US ARMY ARMAMENT RESEARCH, DEVELOPMENT AND ENGINEERING CENTER
CLOSE COMBAT ARMAMENTS CENTER
BENÉT LABORATORIES
WATERVLIET, N.Y. 12189-4050

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Utilization of Ion Chromatography and Statistics to Determine Important Acids in Chromium Plating and Electropolishing Solutions

Sam Sopok

Benét Laboratories

Watervliet, NY 12189

Abstract

Inadequate monitoring of sulfuric and chromic acids in chromium plating solutions and phosphoric and sulfuric scids in electroplolishing solutions can cause serious problems for the chromium plating industry, such as poor quality products, wasted human resources, and wasted electrical energy. Current instrumental analysis methods, both offline and online, for these acids either do not result in acceptable precisions or are time consuming. Two similar methods, which are improvements on existing ion chromatographical methods, are presented here that provides acceptable analysis and monitoring of these acids in this chromium plating process.

Keywords

instrumental analysis, offline monitoring, online monitoring, sulfuric acid, chromic acid, phosphoric acid, chromium plating solutions, electropolishing solutions, ion chromatography, statistics

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Introduction

Inadequate monitoring of sulfuric and chromic acids in chromium plating solutions and phosphoric and sulfuric acids in electroplolishing solutions can cause serious problems for the chromium plating industry, such as poor quality products, wasted human resources, and wasted electrical energy. Current instrumental analysis methods, both offline and online, for these acids either do not result in acceptable precisions or are time consuming (ref 1-5). Two similar methods, which are improvements on existing ion chromatographical methods, are presented which provide acceptable analysis and monitoring of these acids in this chromium plating process coupled with an extensive statistical evaluation of the experimental data.

Approach

Strict analytical chemistry methods and procedures are followed throughout this experimental section. An excellent source of reference for these methods and procedures is by Fritz and Schenk (ref 6).

The analytical system used is the Dionex 2020i ion chromatograph (Dionex Corporation, Sunnyvale, CA). Dionex publishes a manual which is an excellent source of reference for operation and maintenance of this chromatographic system (ref 7).

The initial conditions for the autosampler module, analytical pump, conductivity detector, and advanced chromatography modules are given in Tables 1-4, respectively.

Fill the system reservoirs with the appropriate method eluent and regenerate. The analytical pump used for the eluent must be completely purged of air every time the eluent reservoir is filled. Dionex claims that a proper functioning analytical pump is necessary for acceptable precisions.

The conductivity detector should be periodically calibrated to 147 μS using 0.00100 M potassium chloride.

The initial programing conditions for the system computer/controller module are the equilibration program (P-1), analysis program (P-2), and the halt program (P-H) given in Tables 5-7, respectively. The initial scheduling conditions for this same module for the equilibration, analysis, and halt programs are given in Table 8.

The initial programing and scheduling conditions for the system computer/integrator are given in Table 9.

Determination of chromic or sulfuric acid in chromium plating solutions

Two analytical reagent grade standard solutions are required. The first is a 2.95 ± 0.01 g/l sulfuric acid solution that meets American Chemical Society (ACS) and American Society For Testing Materials (ASTM) Standards (ref 8-9). The second is a 250 ± 1 g/l chromic acid solution that meets ACS and Federal Standards (ref 10-11). E. M. Science reagent grade chromium trioxide is the only material found in our experience that meets ACS standards allowing a maximum of 0.005% sulfate. Two other reagent grade solutions are also required: a 0.38 ± 0.02 g/l sodium carbonate eluent solution and a 1.38 ± 0.04 g/l sulfuric acid regenerate solution.

Preparation of a chromium plating solution standard for ion chromatographic analysis requires that one milliliter of each of the analytical reagent grade standard solutions (sulfuric and chromic acids) prepared above are diluted to the mark with deionized water in a 250 ml volumetric flask. Only two chromium plating solution samples (X & Y) can be run at a time since each of these two sample solutions are split for a total of four solutions (X-1, X-2, Y-1, and Y-2).

Preparation of a chromium plating solution sample for ion chromatographic analysis requires that one milliliter of each split solution is diluted to the mark with deionized water in a 250 ml volumetric flask.

The chromium plating solution standard (S) and samples (2-1, 2-2, 8-1, and 8-2) for ion chromatographic analysis are placed in the autosampler cassettes in thirty sealed 5 ml vials according to the order outlined in Table 10. Using the same order, these cassettes are placed in the autosampler module.

A chromatograph is produced for each chromium plating solution standard (S) and samples (2-1, 2-2, 8-1, and 8-2) placed in the autosampler for analysis according to the order outlined in Table 10. For these standard and sample solutions, peak heights are given for the acid and since standard solution concentrations are known, sample solution concentrations can be calculated. Then precisions are calculated using an extensive statistical method for reliability determinations.

Determination of sulfuric or phosphoric acid in electropolishing solutions

One analytical reagent grade standard solution is required. It contains 685 ± 1 g/l phosphoric acid and 845 ± 1 g/l sulfuric acid. This standard solution is prepared and standardized using a previous method by this author (ref 12). Two other reagent grade solutions are required: a 0.100 ± 0.005 g/l sodium carbonate eluent solution and a 1.38 ± 0.04 g/l sulfuric acid regenerate solution.

Preparation of a electropolishing solution standard for ion chromatographic analysis requires that 0.400 ml of the analytical reagent grade standard solution (phosphoric and sulfuric acid mixture) prepared above is diluted to the mark with deionized water in a 2000 ml volumetric flask. Up to two electropolishing solution samples (X & Y) can be run at a time since each of these two sample solutions are split for a total of four solutions (X-1, X-2, Y-1, and Y-2). Only one sample solution is run (8P) here which is split into solutions 8P-1, and 8P-2.

Preparation of a electropolishing solution sample for ion chromatographic analysis requires that 0.400 ml of the split solution is diluted to the mark with deionized water in a 2000 ml volumetric flask.

The electropolishing solution standard (S) and samples (8P-1 and 8P-2) for ion chromatographic analysis are placed in the autosampler cassettes in 18 sealed 5 ml vials according to the order outlined in Table 11. Using the same order, these cassettes are placed in the autosampler module.

A chromatograph is produced for each electropolishing solution standard (S) and samples (8P-1 and 8P-2) placed in the autosampler for analysis according to the order outlined in Table 11. For these standard and sample solutions, peak heights are given for the acid and since standard solution concentrations are known, sample solution concentrations can be calculated. Then precisions are calculated using an extensive statistical method for reliability determinations.

Results and Discussion

A statistical analysis is necessary to determine the reliability of the experimental ion chromatographic sample data in order to adequately monitor a given acid in the chromium plating process. The statistical evaluation has two parts.

In the first part of the statistical evaluation, samples are split and each split solution is analyzed in triplicate. Tables 12 and 13 give the experimental peak height data for the acid standard and split sample solutions.. The mean X(avg) and sample standard deviation S(n-1) are calculated for each of these samples and its associated standard. The pooled sample standard deviation Sp is calculated from the standard deviation of each sample and its associated standard as follows:

$$Sp = \left[\frac{(S1^{**2} + S2^{**2})}{2} \right]^{**0.5} \tag{1}$$

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In addition, the ninety-five percent confidence level C(95%) is calculated from the above quantities for each sample and its associated standard:

$$C(95\%) = (2.266)(Sp)\frac{\text{acid std. conc}}{\text{std. X(avg)}} \qquad (2)$$

The concentration data are given for the experimental peak height data in Tables 14 and 15. Again, the mean X(avg) and sample standard deviation S(n-1) are calculated for each of these samples.

The concentration data of the split sample solutions are given in Table 16 and 17. As above, the mean, sample standard deviation, and pooled sample standard deviation are calculated. In addition, another quantity is calculated called the T-test value (T) for each sample solution from the following:

$$T = (1.225)\frac{X1(\text{avg})-X2(\text{avg})}{Sp} \qquad (3)$$

The experimental data of the split solutions of a sample solution are compared to a 2.776 value using the T-test to see if they are statistically from the same original sample solution to a 95% confidence level. A sample solution passes this T-test if its resultant absolute value is less than the 2.776 value. This T-test addresses the reliability dilemma mentioned above since it either accepts or rejects a sample solution analysis; samples solutions that fail the T-test are statistically dissimilar and are re-sampled and re-analyzed.

In the second part of the statistical evaluation, the mean X(avg) and normal population standard deviation (Sn) are calculated for the six respective replicates of the sample solutions that have passed the T-test. Since this was the case, the concentration data for the solutions are given in Table 18 and 19.

It is useful to evaluate the variations in precision for the materials and methods used. Tables 20-23 present the data for the one milliliter micropipet, the 250 ml class A volumetric flasks, the 250 g/l chromic acid standard solution and the 2.95 g/l sulfuric acid standard solution, respectively. Variations in precision are also evaluated for the ion chromatographic system. Table 24 and 25 present the data for 24 consecutive replicates of the 2.95 g/l sulfuric acid and 2.50 g/l chromic acid standard solutions which are equivalent to the same number of runs as analyzing the split solutions of sample solutions 2 and 8. Tables 26-29 present the data for the 0.400 ml micropipet, the 2000 ml class A volumetric flask, the 685 g/l phosphoric acid standard solution and the 845 g/l sulfuric acid standard solution, respectively. Variations in precision are also evaluated for the ion chromatographic system. Table 30 and 31 presents the data for 6 consecutive replicates of the 685 g/l phosphoric acid and 845 g/l sulfuric acid

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standard solutions. It is clear that the ion chromatographic system contributes the greatest amount of variation in precision compared to all other sources given for both of the above methods.

The data shows that these improved methods are sufficient to monitor these acids in the chromium plating process. The optimum operating range of sulfuric acid in chromium plating solutions is 2.50 - 3.00 g/l and the resulting precisions are in the range of 0.03 - 0.20 g/l. The optimum operating range of chromic acid in chromium plating solutions is 240 - 260 g/l and the resulting precisions are in the range of 1 - 9 g/l. The optimum operating range of phosphoric in electropolishing solutions is 640 - 730 g/l and the resulting precisions are in the range of 2 - 7 g/l. The optimum operating range of sulfuric acid in electropolishing solutions is 795 - 895 g/l and the resulting precisions are in the range of 2 - 8 g/l. These precisions provide adequate monitoring of these chromium plating process acids and are supported by eight years of testing.

Table 1. Initial Conditions for the Autosampler Module

Local/Remote:	Local
Run/Hold:	Hold Then Run
Status:	Search
Size:	5 ml
Type:	Sample
Inj:	1
Tray:	In/Empty
Type:	Loop
Mode:	Prop
Bleed:	On
Inj/Vial:	1

Table 2. Initial Conditions for the Analytical Pump Module

Local/Remote:	Remote
Start/Stop:	Stop
Flow (ml/min):	2.3
Pressure Limit Select Low Alarm (psi):	100
Pressure Limit Select High Alarm (psi):	1000
Valve Pressure (psi Argon):	100

Table 3. Initial Conditions for the Conductivity Detector Module

Local/Remote:	Remote
Cell:	On
Auto Offset:	Off
Temp. Comp.:	0
Output Range (μS):	30

Table 4. Initial Conditions for the Advanced Chromatography Module

Load/Inject:	Inject
Valve A:	Off
Valve B:	Off
Local/Remote:	Remote
Separator Column:	HPIC-AG4 (Dionex)
Suppressor Column:	AMMS (Dionex)
Valve Pressure (psi Argon):	100
Regenerate Flow:	5ml/min
Regenerate Pressure (psi Argon):	6

Table 5. Computer/Controller Equilibration Program One*

Time (min)	Load/Inject	Valve B	Offset	Auto-Relays	End Run
0.0	Inject	On	Off	Off	Off
5.0	Inject	On	Off	Off	Off
30.0	Inject	On	Off	Off	On

Table 6. Computer/Controller Analysis Program Two*

Time (min)	Load/Inject	Valve B	Offset	Auto-Relays	End Run
0.0	Load	On	Off	Off	Off
0.1	Load	On	Off	#2	Off
0.2	Load	On	Off	Off	Off
2.2	Load	On	On	Off	Off
2.3	Inject	On	On	#1	Off
10.0	Inject	On	On	Off	On

* Eluent flow rate is 2.3 ml/min., eluent port #3 is used, temp. select is zero, valve A is continually off, temp. comp. is 1.7, cond. setting full scale is 30 µS, AC outlets are all continually off.

Table 7. Computer/Controller Halt Program Halt

Time (min)	Load/Inject	Valve B	Offset	Auto-Relays	End Run
0.0	Inject	Off	Off	Off	On

Eluent flow rate is 0.0 ml/min., no eluent port is selected, temp. select is zero, valve A is continually off, temp. comp. is zero, cond. setting full scale is 30 µS, AC outlets are all off.

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Table 8. Computer/Controller Analysis Schedule

Step	Program	Iterations
1	1	1
2	2	30
3	Halt	1
4-9	Halt	0

Table 9. Computer/Integrator Analysis Program

Ready

Date"

Time"

AT=1024

PH=1

PT=5000

FI=1. FE=1. MN=0.

Press 'Enter' To Skip Entry

File Name="

Time Function Value

TT=

Method Number: MN=0

End of Dialog

Table 10. Order of Sample Vials in the Autosampler Cassettes for Sulfuric and Chromic Acid in Chromium Plating Solution

Sample Vial #	Standard/Sample #
1	S
2	S
3	S
4	S
5	S
6	2-1
7	S
8	2-1
9	S
10	2-1
11	S
12	2-2
13	S
14	2-2
15	S
16	2-2
17	S
18	8-1
19	S
20	8-1
21	S
22	8-1
23	S
24	8-2
25	S
26	8-2
27	S
28	8-2
29	S
30	Deionized Water

Table 11. Order of Sample Vials in the Autosampler Cassettes for Phosphoric and Sulfuric Acid in Electropolishing Solution

Sample Vial #	Standard/Sample #
1	S
2	S
3	S
4	S
5	S
6	8P-1
7	S
8	8P-1
9	S
10	8P-1
11	S
12	8P-2
13	S
14	8P-2
15	S
16	8P-2
17	S
18	Deionized Water

Table 12. Experimental Ion Chromatograph Peak Height Data for Sulfuric and Chromic Acid in Chromium Plating Sample Solutions

Replicate	Sulfuric Acid Standard S	Sample 2-1	Standard S	Sample 2-2	Chromic Acid Standard S	Sample 2-1	Standard S	Sample 2-2
1	185033	165973	180188	168167	638810	666837	647006	672091
2	168196	159359	171808	162845	680478	663962	683074	675875
3	170185	169031	168119	165749	685812	689280	694193	669281
X(avg)	174471	164788	173372	165587	668366	673359	674757	672415
S(n-1)	9200	4944	6184	2645	25739	13862	25668	3308
Sp	8171		4756		20671		17599	
C(95%)	0.312		0.182		17.5		14.8	

Replicate	Sulfuric Acid Standard S	Sample 8-1	Standard S	Sample 8-2	Chromic Acid Standard S	Sample 8-1	Standard S	Sample 8-2
1	170185	163899	173776	163668	667793	678899	678874	675566
2	176065	165987	169989	163303	671066	682189	688216	693419
3	173267	159973	170932	161416	672645	687026	652000	686209
X(avg)	173172	163286	171566	162796	670501	682704	673030	685064
S(n-1)	2941	3053	1971	1209	2474	4087	18801	8981
Sp	2998		1635		3378		14733	
C(95%)	0.115		0.063		2.9		12.4	

Raw peak height data is in integrator units (IC). Initially four equilibration standards are run before standard one. These are standard solutions (S) and split sample solutions (2-1, 2-2, 8-1 and 8-2) after a 1:250 dilution.

Table 13. Experimental Ion Chromatograph Peak Height Data for Phosphoric and Sulfuric Acid in Electropolishing Sample Solutions

Replicate	Phosphoric Acid				Sulfuric Acid			
	Standard S	Sample 8P-1	Standard S	Sample 8P-2	Standard S	Sample 8P-1	Standard S	Sample 8P-2
1	562500	509200	573900	512700	813800	770200	815700	762300
2	572000	507700	575800	516100	821400	764600	813600	763200
3	573300	509100	578700	517100	819500	763000	810400	760500
X(avg)	569267	508667	576133	515300	818233	765933	813233	762000
S(n-1)	5896	839	2417	2307	3955	3781	2669	1375
Sp	4211		2363		3869		2123	
C(95%)	11.5		6.4		9.1		5.0	

Raw peak height data is in integrator units (IU). Initially four equilibration standards are run before standard one. These are standard solutions (S) and split sample solutions (8P-1 and 8P-2) after a 1:5000 dilution.

Table 14. Concentration Data for Sulfuric and Chromic Acid in Chromium Plating Sample Solutions

Replicate	Sulfuric Acid				Chromic Acid			
	Sample 2-1	Sample 2-2	Sample 8-1	Sample 8-2	Sample 2-1	Sample 2-2	Sample 8-1	Sample 8-2
1	2.63	2.74	2.83	2.76	261	260	254	249
2	2.78	2.78	2.77	2.82	244	247	254	252
3	2.92	2.89	2.71	2.77	251	241	255	263
X(avg)	2.77	2.80	2.77	2.78	252	249	254	255
S(n-1)	0.145	0.078	0.060	0.032	8.54	9.71	0.577	7.37

Concentration data is in g/l.

Table 15. Concentration Data for Phosphoric and Sulfuric Acid in Electropolishing Sample Solutions

Replicate	Phosphoric Acid		Sulfuric Acid	
	Sample 8P-1	Sample 8P-2	Sample 8P-1	Sample 8P-2
1	620	612	800	790
2	608	614	787	793
3	608	612	787	793
X(avg)	612	613	791	792
S(n-1)	6.9	1.1	7.5	1.8

Concentration data is in g/l.

Table 16. Concentration Data for Sulfuric and Chromic Acid in Chromium Plating Sample Solutions

Split Solutions	Sulfuric Acid				Chromic Acid			
	Sample Solution 2		Sample Solution 8		Sample Solution 2		Sample Solution 8	
	2-1	2-2	8-1	8-2	2-1	2-2	8-1	8-2
X(avg)	2.77	2.80	2.77	2.78	252	249	254	255
S(n-1)	0.145	0.078	0.060	0.032	8.54	9.71	0.577	7.37
Sp	0.116		0.048		9.14		5.22	
T-test value (T)	0.316		0.254		0.402		0.234	

Concentration data is in g/l. These are the split sample solutions (2-1, 2-2, 8-1 and 8-2) from original sample solutions 2 and 8.

Table 17. Concentration Data for Phosphoric and Sulfuric Acid in Electropolishing Sample Solutions

Split Solutions	Sample Solution 8P			
	Phosphoric Acid		Sulfuric Acid	
	8P-1	8P-2	8P-1	8P-2
X(avg)	612	613	791	792
S(n-1)	6.9	1.1	7.5	1.8
T-test value (T)	0.13		0.17	

Concentration data is in g/l.

Table 18. Reported Concentration Data for Sulfuric and Chromic Acid in Chromium Plating Sample Solutions

Replicate	Sulfuric Acid		Chromic Acid	
	Sample Solution 2	Sample Solution 8	Sample Solution 2	Sample Solution 8
1	2.63	2.83	261	254
2	2.78	2.77	244	254
3	2.92	2.71	251	255
4	2.74	2.76	260	249
5	2.78	2.82	247	252
6	2.89	2.77	241	263
X(avg)	2.79	2.77	251	254
Sn	0.10	0.04	8	4

Concentration data is in g/l. Sample solutions 2 and 8 are the unsplit and original sample solutions.

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Table 19. Reported Concentration Data for Phosphoric and Sulfuric Acid in Electropolishing Sample Solutions

	Phosphoric Acid	Sulfuric Acid
Replicate	Solution 8P	Solution 8P
1	620	800
2	608	787
3	608	787
4	612	790
5	614	793
6	612	793
X(avg)	612	792
Sn	4.0	4.5

Concentration data is in g/l. Sample solution 8P is the unsplit and original sample solution.

Table 20. Precision of Micropipeting One Milliliter

Replicate	Volume (ml)
1	1.0133
2	1.0105
3	1.0140
4	1.0066
5	1.0106
6	1.0065
X(avg)	1.0102
Sn	0.0029

Table 21. Precision of 250 ml Class-A Volumetric Flask

Replicate	Volume (ml)
1	249.54
2	249.47
3	249.47
4	249.45
5	249.50
6	249.51
X(avg)	249.49
Sn	0.03

Volumes are calculated from the weight-volume relationship of the contained deionized water solution corrected for temperature.

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Table 22. Precision of 250 g/l Chromic Acid Standard Solution

Replicate	CRO$_3$ Conc. (g/l)
1	250.5
2	250.2
3	250.2
4	249.8
5	250.5
6	250.2
X(avg)	250.2
Sn	0.2

Chromic acid concentrations are calculated using Federal Specification O-C-303D.

Table 23. Precision of 2.95 g/l Sulfuric Acid Standard Solution by Titration

Replicate	H$_2$SO$_4$ Conc. (g/l)
1	2.940
2	2.945
3	2.940
4	2.945
5	2.935
6	2.945
X(avg)	2.942
Sn	0.004

Sulfuric acid concentrations are calculated using ASTM Standard E233.

Table 24. Precision of 2.94 g/l Sulfuric Acid Standard Solution by Ion Chromatography

Replicate	H_2SO_4 Peak Height (IU)
1	190949
2	188708
3	190668
4	190147
5	190088
6	192674
7	188182
8	187171
9	185994
10	185739
11	188327
12	191380
13	190464
14	185171
15	185513
16	184750
17	189062
18	188087
19	189516
20	185360
21	186953
22	182093
23	187845
24	183962
X(avg)	187866
Sn	2650

Table 25. Precision of 250 g/l Chromic Acid Standard Solution by Ion Chromatography

Replicate	$CRO3$ Peak Height (IU)
1	686587
2	647711
3	659666
4	675259
5	689793
6	699663
7	682745
8	680179
9	686991
10	674796
11	693042
12	703530
13	693399
14	690266
15	684613
16	683087
17	693316
18	698195
19	699618
20	697099
21	677752
22	663899
23	705008
24	680866
X(avg)	685295
Sn	14035

Peak height is in integrator units (IU). Four equilibration replicates were run before the first replicate in this Table; these twenty-four replicates represent the same number of samples run as when sample solutions 2 and 8 are split and run above.

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Table 26. Precision of Micropipeting 0.400 Milliliters

Replicate	Volume (ml)
1	0.401
2	0.398
3	0.399
4	0.400
5	0.400
6	0.403
X(avg)	0.400
Sn	0.002

Table 27. Precision of 2000 ml Class-A Volumetric Flask

Replicate	Volume (ml)
1	2002
2	1997
3	1997
4	2001
5	2003
6	1998
X(avg)	2000
Sn	2

Volumes are calculated from the weight-volume relationship of the contained deionized water solution corrected for temperature.

Table 28. Precision of 685 g/l Phosphoric Acid Standard Solution

Replicate	H_3PO_4 Conc. (g/l)
1	684
2	685
3	685
4	684
5	685
6	684
X(avg)	685
Sn	1

Table 29. Precision of 845 g/l Sulfuric Acid Standard Solution by Titration

Replicate	H_2SO_4 Conc. (g/l)
1	845
2	845
3	846
4	845
5	844
6	845
X(avg)	845
Sn	1

Phosphoric acid concentrations are calculated by a chemical analysis method by this author (12).

Sulfuric acid concentrations are calculated by a chemical analysis method by this author (12).

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Table 30. Precision of 685 g/l Phosphoric Acid Standard Solution by Ion Chromatography	
Replicate	H_3PO_4 Peak Height (IU)
1	562500
2	572000
3	573300
4	573900
5	575800
6	578700
X(avg)	572700
Sn	5032

Table 31. Precision of 845 g/l Sulfuric Acid Standard Solution by Ion Chromatography	
Replicate	H_2SO_4 Peak Height (IU)
1	813800
2	821400
3	819500
4	815700
5	813600
6	810400
X(avg)	815700
Sn	3720

Peak height is in integrator units (IU). Four equilibration replicates were run before the first replicate in this Table.

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